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AD NUMBER
ADB064672
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AD-E400 823

TECHNICAL REPORT ARSCD-TR-82003

ADHESIVE BONDING OF OILY STEEL: SHEAR STRENGTH STUDIES

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APRIL 1982

U.S. ARMY ARRADCOM

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND

FIRE CONTROL AND SMALL CALIBER WEAPON SYSTEMS LABORATORY

DOVER, NEW JERSEY

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER Technical Report ARSCD-TR-82003	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) ADHESIVE BONDING OF OILY STEEL: SHEAR STRENGTH STUDIES		5. TYPE OF REPORT & PERIOD COVERED Final
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Roberta Rosty, Robert B. Bonk William Russell, David W. Levi		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS ARRADCOM, FC&SCWSL Mat'ls & Mfg Tech Div (DRDAR-SCM) Dover, NJ 07801		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS AMCMS 612105H840011
11. CONTROLLING OFFICE NAME AND ADDRESS ARRADCOM, TSD STINFO DIV (DRDAR-TSS) Dover, NJ 07801		12. REPORT DATE April 1982
		13. NUMBER OF PAGES 39
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Distribution limited to US Government agencies only; test and evaluation; April 1982. Other requests for this document must be referred to ARRADCOM, ATTN: DRDAR-TSS, Dover, NJ 07801.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Steel
Adhesive bonding of steel
Oily steel
Adhesive bonding of oily steel
Shear strength
Mechanical properties

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Ten promising commercial adhesives were evaluated with respect to their ability to bond to oily steel. Nine of them formed initial bonds with some oily steel surfaces that compared favorably in shear strength with control specimens (vapor degreased and solvent wiped steel). The specific value of the strength was found to be highly dependent on the adhesive-oil system used. This work suggests the possibility of cleaning steel surfaces by washing with an oil, followed by bonding with an adhesive that is compatible with the particular (cont)

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20. Abstract (cont)
oil. No durability work has yet been conducted on these adhesive bonded joint systems.

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INTRODUCTION

It would be desirable in the adhesive bonding of steel in many munition items if the surface preparation process for the steel could be simplified or even eliminated. The safe use of solvents and chemicals on a production line in cleaning the steel is often tedious, troublesome and expensive. A number of modern adhesives may have some capacity for absorbing the oils that are found on steels, with the resulting tendency to lift the oil and bond to the surface underneath. Unfortunately the data on bonding these adhesives to oily steel tends to be scattered and fragmentary.

In the present work ten commercial adhesives with the potential for bonding oily steel have been selected. Shear strengths of the adhesive bonds to steel have been measured after each of the following treatments:

1. Controls - The steel panels were vapor degreased. After vapor degreasing, a considerable amount of dirt was still evident on the surface. Therefore, the steel panels were additionally acetone wiped (Set 1).

2. As-received - The steel panels were lightly wiped with a tissue to remove excess oil and surface dirt (Set 2).

3. The as-received steel panels were acetone wiped and then lightly coated with a water soluble oil (Set 3).

4. The as-received panels were acetone wiped and then lightly coated with a non-water soluble oil (Set 4).

This report gives results of the adhesive bond shear strength study for steel panels treated by each of the four methods.

RESULTS AND DISCUSSION

The raw shear strength data for the ten adhesives are shown in tables 1 through 10. Cursory examination of these tables indicates that 9 of the 10 adhesives form bonds that show very appreciable strengths with all of the oily steel Sets, in some cases even exceeding the controls. This examination also seems to indicate that the exact behavior is highly dependent on the adhesive and oil combination used.

In order to more carefully examine the results for the individual adhesives, a graphical representation of the data is desirable. For this purpose, the Weibull distribution has been found to be useful for

this type of mechanical data (refs. 1 and 2).

For the present purpose the cumulative distribution function may be written (refs. 3 and 4):

$$\log \log \left[\frac{1}{1-F(x)}\right] = -\log \alpha + \beta \log(X-\gamma) \quad (1)$$

where F(x) is the distribution function, i.e., the fraction of samples showing a shear strength value of X or below. α is the scale parameter (y intercept), β is the slope and γ is a location or threshold parameter. A plot of the left hand side of equation (1) versus $\log(X-\gamma)$ should give a straight line of slope β and intercept α. γ may be selected on an iterative basis by making trial plots. In the present work satisfactory results were obtained by taking $\gamma = 0$ and thus using a two parameter distribution.

The Wilcoxon Sum of Ranks test was used where necessary to determine whether two (or more) sets of data for a given adhesive were equivalent. This statistical test is rapid and convenient and has the advantage that it requires no assumption concerning the distribution of the data (ref. 5).

Versilok 202, Versilok 204, Loctite "Depend" and Dymax 845

The commercial adhesives Versilok 202, Versilok 204, Loctite "Depend" and Dymax 845 showed a somewhat similar pattern of behavior in this study. Figures 1 through 4 graphically illustrate this point. In each case the strongest bonds are shown by the controls (Set 1) and the Dore water soluble oil treatment (Set 3). The data for Set 1 and Set 3 in all four cases are indistinguishable. This indicates that this oil left on the steel surface would give initial bonds just as good as a conventionally cleaned surface (controls), a very interesting and potentially important observation.

For these adhesives systems, Set 2 (as-received steel) and Set 4 (non-water soluble oil Kwik Kut) gave somewhat lower shear strengths, although usable bonds were still formed. As shown in Figures 1 and 2, for Versilok 202 and Versilok 204, Sets 2 and 4 gave the same strength. However, for Loctite "Depend" and Dymax 845 the Set 2 was noticeably lower in strength than Set 4. The magnitude of these differences can be seen in Figures 1 through 4.

Versilok 202, Versilok 204 and Loctite "Depend" are acrylic type adhesives while Dymax 845 is not identified. For this group we can conclude that the Dore water soluble oil on the surface does not interfere with adhesive bonding. With Kwik Kut oil on the surface, bonds are formed but at a somewhat lower strength. Presumably the

rolling oil and dirt on the surface of as-received steel interferes with bonding to some extent, as reflected in the lower strengths. Just how much lower this strength is seems to depend on the particular adhesive utilized.

It should be remarked at this point that not all of the acrylics studied behaved as described for the ones above. These differences will become evident in the later discussion.

Versilok 200

Although the Versilok 200 is an acrylic, its behavior toward oily steel is markedly different from the group discussed above. Both Set 2 (as-received) and Set 4 (Kwik Kut non-water soluble oil) broke while preparing specimens so that no shear strength data could be obtained.

Figure 5 shows that Set 3 (Dore water soluble oil) gave significantly greater strength than the controls. Two sets of controls were tested and there was a significant difference between them as indicated in Figure 5. Since this adhesive did not appear to be promising in bonding oily steel (except for Set 3), it was not studied further.

Plastilock A-1

The Plastilock adhesive is a modified structural acrylic that behaves somewhat differently with oily steel than the other acrylics. This behavior is shown in Figure 6. In this case both Set 3 (Dore water soluble oil) and Set 4 (Kwik Kut non-water soluble oil) show a small but significant increase in shear strength when compared with the controls (Set 1). Furthermore, Sets 3 and 4 are statistically indistinguishable. Set 2 (as-received) gives markedly lower data than any of the others. This emphasizes once again the highly specific behavior of the various adhesive-oil combinations.

Cybond 4533, Epibond 1210 and Epon 828/Epon Curing Agent V-40

The epoxides Epibond 1210 and Epon 828/Epon curing agent V-40 are the only adhesives studied that form bonds with the as-received steel (Set 2) that are equivalent to the controls (Set 1). This is clearly shown in Figures 7 and 8. The as-received steel bond with the Cybond 4533 is somewhat inferior to the controls but the difference is not great and there is a considerable data overlap as shown in Figure 9. In other respects the epoxides tend to be somewhat erratic in their behavior. For example, the Dore water soluble oil (Set 3) forms inferior bonds with the Cybond and Epibond but it is equivalent

to the controls (Set 1) for the Epon. The Kwik Kut non-water soluble oil (Set 4) is inferior with the Epon and Epibond but the same as the controls (Set 1) for the Cybond.

Conastic 830

Results for the Conastic 830, which was not identified as to chemical type, are shown in Figure 10. In this case the controls (Set 1) and both applied oils (Sets 3 and 4) showed identical shear strengths. The strengths to the as-received steel (Set 2) were markedly lower.

Results

The results in this preliminary report indicate that it is possible to bond to oily steel without a preliminary surface cleaning. It appears that particular adhesive-oil systems are highly specific, so that care must be taken in selecting the adhesive to bond to a particular surface. Further work along this line is needed. In addition, a study of the durability of bonds to the different surfaces seems indicated before any practical applications can be undertaken.

It appears probable that good bonds are obtained when the adhesive absorbs the oil and then bonds to the surface underneath. In certain cases these bonds may actually be stronger than those to chemically clean surfaces due to the plasticizing effect of the oil on the adhesive. This suggests that some steel surfaces might be treated by washing them with an oil that is compatible with the adhesive to be used. In this way the use of troublesome and/or hazardous cleaning solvents and chemicals may be avoided. Additional work toward this end is needed.

EXPERIMENTAL PROCEDURES

Materials

Steel	A 1020 cold rolled steel alloy was used. It had the following ladle composition limits: C, 0.17 - 0.24%; Mn, 0.3 - 0.6%; P (max), 0.04%; S (max), 0.05%.
Adhesives	The adhesives are shown in table 11.
Oils	The oils are given in table 12.

Preparation of Steel Panels for Mechanical Fastening

The steel panels (12" x 4") were overlapped along the 12" length for 1/2 inch. One hole was drilled through this overlap at each end for the purpose of holding the bonded panels in alignment while the adhesive was curing.

Steel Surface Preparation

Controls (Set 1)

The controls were vapor degreased and then wiped with acetone soaked paper tissue (Scott Assembly Wipes) to remove all visible traces of dirt.

As-Received Steel (Set 2)

The as-received steel panels have dirt and milling oil on their surfaces. These panels were wiped with dry paper tissues to remove excess oil and dirt.

Dore Oiled Specimens (Set 3)

The as-received steel panels were wiped with acetone soaked paper tissue to remove all visible traces of oil and dirt. The Dore oil was spread over the surface with a tissue soaked in the oil.

Kwik Kut Oiled Specimens (Set 4)

The as-received steel panels were wiped with paper tissues soaked with acetone to remove all visible traces of oil and dirt. The Kwik Kut oil was spread over the surface with a tissue soaked in the oil.

Bonding and Curing

In Sets 2, 3, and 4 the excess oil on the surface was removed with a tissue.

The prepared steel surfaces were primed (or an accelerator was applied if required). If a drying time was specified in the manufacturer's instructions for the primer or accelerator, the specimens were set aside to dry.

The adhesive resin was applied in excess to insure adequate surface coverage and the two steel surfaces were joined. The steel

panels were fixtured to maintain a 1/2 inch lap joint by hammering small metal plugs in the holes, drilled as described above. Weights were placed in the center of the panels to assure even contact along the length of the panel. The bonded panels were cured according to the specifics in table 13.

Cutting and Testing the Steel Specimens

The steel specimens were cut 1" wide and the lap shear test was performed in accordance with ASTM-D-1002 using a 1" wide by 1/2" long lap shear joint loaded at a rate of 2400 psi per minute. Testing was at 70 - 73°F on a Baldwin test machine.

CONCLUSIONS

1. It is possible to form strong preliminary adhesive bonds to oily steel.

2. Particular adhesives are highly selective with different oils with respect to the strength of the resultant bonds.

3. The results suggest that it may be possible to clean steel surfaces with an oil that is compatible with the adhesive to be used. More work along this line is needed.

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Table 1. Shear strength data for Plastilock adhesive A-1

Shear strength (psi)			
Set 1	Set 2	Set 3	Set 4
1140	1080	2040	2010
1905	1120	2100	2090
1910	1170	2130	2160
1930	1230	2190	2180
1930	1390	2280	2280
1930	1420	2360	2340
1940	1580	2410	2360
1990	1580	2520	2390
2020	1650	2820	2670
2060	1700	2930	2800
1880 = Mean	1390 = Mean	2380 = Mean	2330 = Mean
263 = SD	232 = SD	304 = SD	248 = SD

Set 1 - Controls

Set 2 - As-received steel

Set 3 - With Dore water soluble oil

Set 4 - With Kwik Kut non-water soluble oil

Table 2. Shear strength data for Versilok 202 adhesive

Shear strength (psi)

Set 1	Set 2	Set 3	Set 4
1190	570	1270	120
1410	630	1670	610
1540	920	1780	860
1590	1000	1800	980
1740	1020	1810	990
1880	1050	1970	1010
2010	1070	2000	1150
2160	1160	2010	1160
2250	1270	2040	1250
2590	1380	2050	1320
1840 = Mean	1010 = Mean	1840 = Mean	945 = Mean
426 = SD	253 = SD	239 = SD	354 = SD

Set 1 - Controls

Set 2 - As-received steel

Set 3 - With Dore water soluble oil

Set 4 - With Kwik Kut non-water soluble oil

Table 3. Shear strength data for Versilok 204 adhesive

Shear strength (psi)

Set 1	Set 2	Set 3	Set 4
1570	630	1560	940
1850	1300	2290	1250
1870	1470	2390	1350
2080	1540	2430	1360
2400	1540	2460	1410
2480	1610	2480	1480
2480	1770	2540	1580
2510	1790	2560	1590
2690	1880	2600	1720
2790	2020	2650	1750
2270 = Mean	1560 = Mean	2400 = Mean	1440 = Mean
404 = SD	388 = SD	312 = SD	240 = SD

Set 1 - Controls

Set 2 - As-received steel

Set 3 - With Dore water soluble oil

Set 4 - With Kwik Kut non-water soluble oil

Table 4. Shear strength data for Loctite "Depend" adhesive

Shear strength (psi)

Set 1	Set 2	Set 3	Set 4
1540	360	1180	1380
1630	710	1350	1420
1710	740	1370	1450
1730	850	1470	1480
1860	1050	1550	1530
1910	1160	1630	1570
1920	1250	1970	1580
1980	1390	1980	1630
2070	1390	2080	1680
2150	1530	2290	1720
1850 = Mean	1040 = Mean	1690 = Mean	1540 = Mean
195 = SD	371 = SD	369 = SD	113 = SD

Set 1 - Controls

Set 2 - As-received steel

Set 3 - With Dore water soluble oil

Set 4 - With Kwik Kut non-water soluble oil

Table 5. Shear strength data for Conastic 830 adhesive

Shear strength (psi)			
Set 1	Set 2	Set 3	Set 4
2900	2000	2970	2730
3050	2110	3040	2830
3160	2200	3100	2850
3170	2210	3170	2900
3180	2260	3180	3070
3200	2400	3210	3130
3220	2440	3240	3140
3230	2510	3240	3150
3280	2590	3280	3150
3300	2850	3680	3290
3170 = Mean	2360 = Mean	3210 = Mean	3020 = Mean
117 = SD	252 = SD	191 = SD	182 = SD

Set 1 - Controls

Set 2 - As-received steel

Set 3 - With Dore water soluble oil

Set 4 - With Kwik Kut non-water soluble oil

Table 6. Shear strength data for Epon 828/Epon Curing Agent V-40 adhesive

Shear strength (psi)			
Set 1	Set 2	Set 3	Set 4
1390	1540	1140	350
1460	1550	1540	530
1680	1560	1640	580
1770	1600	1670	820
1820	1630	1690	820
1840	1630	1700	830
1840	1680	1850	900
1930	1710	1930	980
2090	1740	2030	1120
2240	1970	2050	1730
1810 = Mean	1660 = Mean	1720 = Mean	870 = Mean
258 = SD	128 = SD	268 = SD	379 = SD

Set 1 - Controls

Set 2 - As-received steel

Set 3 - With Dore water soluble oil

Set 4 - With Kwik Kut non-water soluble oil

Table 7. Shear strength data for Versilok 200 adhesive

		Shear strength (psi)		
Set 1		Set 2	Set 3	Set 4
Test 1	Test 2			
870	580	Specimens	1140	Panel fell
920	620	fell apart	1150	apart while
1020	710	while	1190	cutting
1020	780	being cut	1370	samples
1030	810		1390	
1090	830		1440	
1140	880		1580	
1230	890		1590	
1250	1130		1610	
1310	1170		1720	
1090 = Mean	840 = Mean		1420 = Mean	
144 = SD	193 = SD		208 = SD	

Set 1 - Controls

Set 2 - As-received steel

Set 3 - With Dore water soluble oil

Set 4 - With Kwik Kut non-water soluble oil

Table 8. Shear strength data for Cybond 4533 adhesive

Shear strength (psi)

Set 1	Set 2	Set 3	Set 4
1840	1090	530	1390
1890	1100	590	1500
1980	1290	680	1500
2000	1620	770	1920
2040	1640	1360	1930
2080	1810	1450	1960
2110	1880	1670	2000
2180	1960	1700	2160
2200	2110	2000	2300
2310	2170	2000	2630
2060 = Mean	1670 = Mean	1280 = Mean	1930 = Mean
144 = SD	394 = SD	583 = SD	387 = SD

Set 1 - Controls

Set 2 - As-received steel

Set 3 - With Dore water soluble oil

Set 4 - With Kwik Kut non-water soluble oil

Table 9. Shear strength data for Epibond 1210 adhesive

Shear strength (psi)

Set 1	Set 2	Set 3	Set 4
1580	1700	1060	1350
1640	2000	1070	1400
1700	2130	1260	1470
1850	2160	1350	1620
1950	2180	1490	1630
2180	2190	1580	1670
2390	2190	1700	1780
2470	2320	2010	2000
2480	2580	2130	2330
2530	2580	2330	2420
2080 = Mean	2200 = Mean	1600 = Mean	1770 = Mean
377 = SD	258 = SD	442 = SD	372 = SD

Set 1 - Controls

Set 2 - As-received steel

Set 3 - With Dore water soluble oil

Set 4 - With Kwik Kut non-water soluble oil

Table 10. Shear strength data for Dymax 845 adhesive

Shear strength (psi)

Set 1	Set 2	Set 3	Set 4
2320	330	2390	1350
2480	450	2520	1350
2490	660	2540	1430
2580	700	2680	1490
2600	900	2800	1650
2600	1050	2800	1720
2600	1060	2810	1730
2620	1390	2840	1990
2680	1440	2870	1990
2900	1450	2890	2590
2590 = Mean	940 = Mean	2710 = Mean	1730 = Mean
149 = SD	407 = SD	173 = SD	383 = SD

Set 1 - Controls

Set 2 - As-received steel

Set 3 - With Dore water soluble oil

Set 4 - With Kwik Kut non-water soluble oil

Table 11. List of adhesives

Manufacturer's Designation	*Curing Component	Type	Manufacturer
Plastilock A-1	Plastilock A-2 primer	Modified structural acrylic	B.F. Goodrich Co., Fabricated Polymers Div., Akron, Ohio
Versilok 200	Accelerator #4	Structural acrylic	Hughson Chem Co., Lord Corp., Erie, PA
Versilok 202	Accelerator #4	Acrylic	Hughson Chem Co., Lord Corp., Erie, PA
Versilok 204	Accelerator #4	Structural acrylic	Hughson Chem Co., Lord Corp., Erie, PA
Depend Adhesive	Depend activator	Base - Elastomer modified methacrylic	Loctite Corp., Newington, Connecticut
Conastic 830 Oil/Grip	Conastic 530 primer	Unknown	Conap, Olean, New York
Epon Resin 828	Epon Curing Agent V-40	Bisphenol epoxy	Shell Chem Co., Distributor: Miller-Stephenson Chem Co., Danbury, CT
Cybond 4533	None - A single component adhesive	Base - Modified epoxy resin	AM Cyanamid Co., Bloomingdale Prod, Havre de Grace, MD
Epibond 1210	Hardener 9610	100% solids - epoxy	M&T Chem, Functional Plastics Div, Los Angeles, CA
Dymax 845 Oil/Grip	530 Activator/primer	Unknown	AM Chem & Engineering Co., Dymax Eng Adhesives, Harwinton, CT

* Hardener, curing agent, primer, or accelerator used

Table 12. List of oils

Manufacturer's Designation	Type	Manufacturer
Dore Heavy Duty Liquid Oil	Water soluble	Dore International Edison, New Jersey
Kwik Kut #1	Not water soluble	Hobart Chem Co., Cedar Grove, New Jersey
Milling oil on as-received steel		

Table 13. Specifics of bonding and curing

Adhesive System	Bonding: Primer or Accelerator used on One or Both Panels		Curing	
	Controls	Oily Surfaces	Temp.	Time
Plastilock A-1/A-2	Both	Both	RT	24 hrs
Versilok 200/Accelerator 4	One	One	RT	4 - 6 min
Versilok 202/Accelerator 4	Both	Both	RT	24 hrs
Versilok 204/Accelerator 4	One	One	RT	24 hrs
Depend Adh/Activator	One	One	RT	4 - 24 hrs
Conastic 830/530 Primer	Both	Both	RT	8 hrs
Epon 828/Epon V-40*	-	-	RT	7 days
Cybond 4533**	-	-	150°C	30 min
Epibond 1210/Hardener 9610*	-	-	RT	48 - 72 hrs
Dymax 845/530 Activator/primer	One	Both	RT	2 - 4 hrs

* Mixed in tin cups with wooden stirrers

** One component system

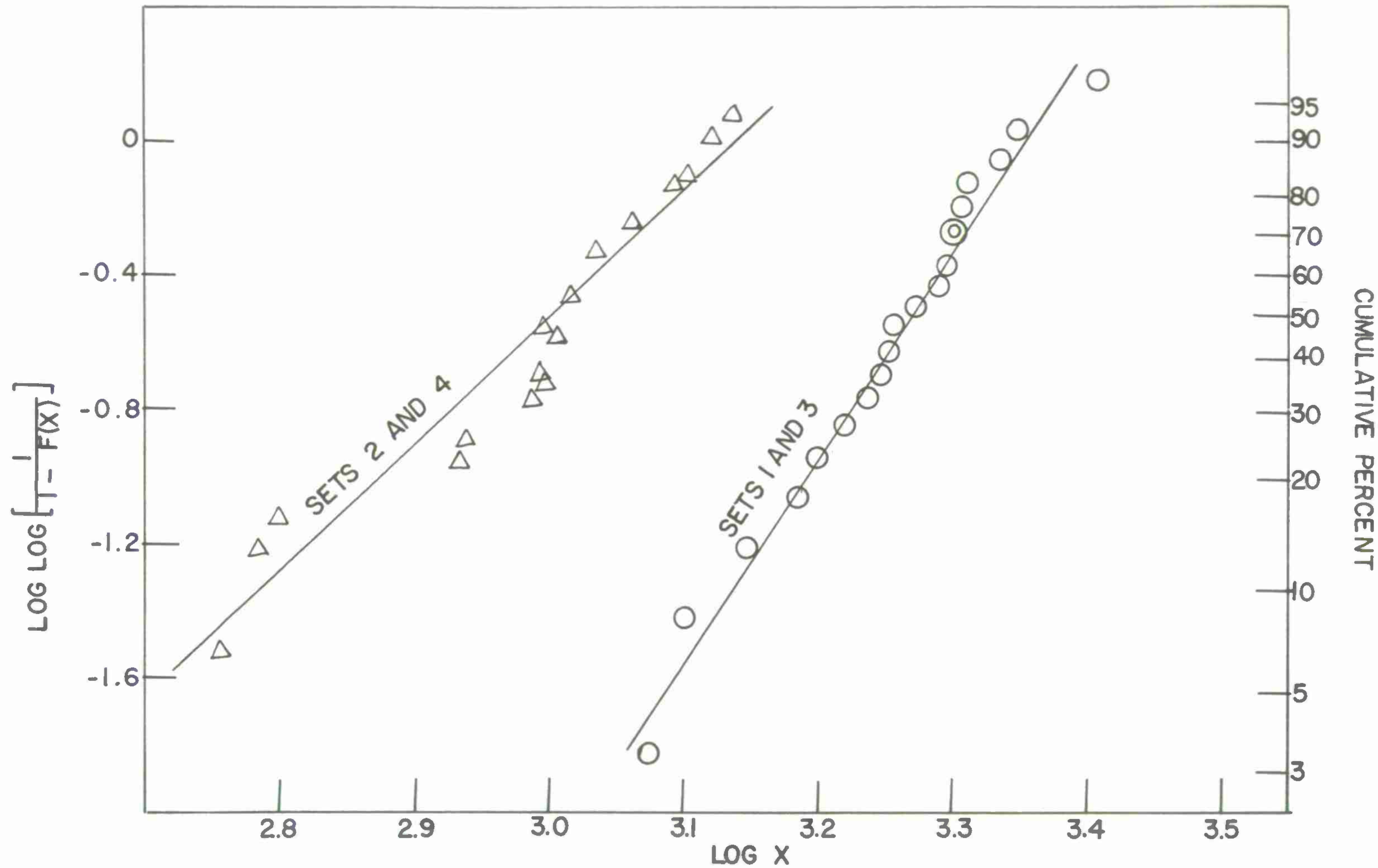

Figure 1. Linear Weibull distribution plots for Versilok 202

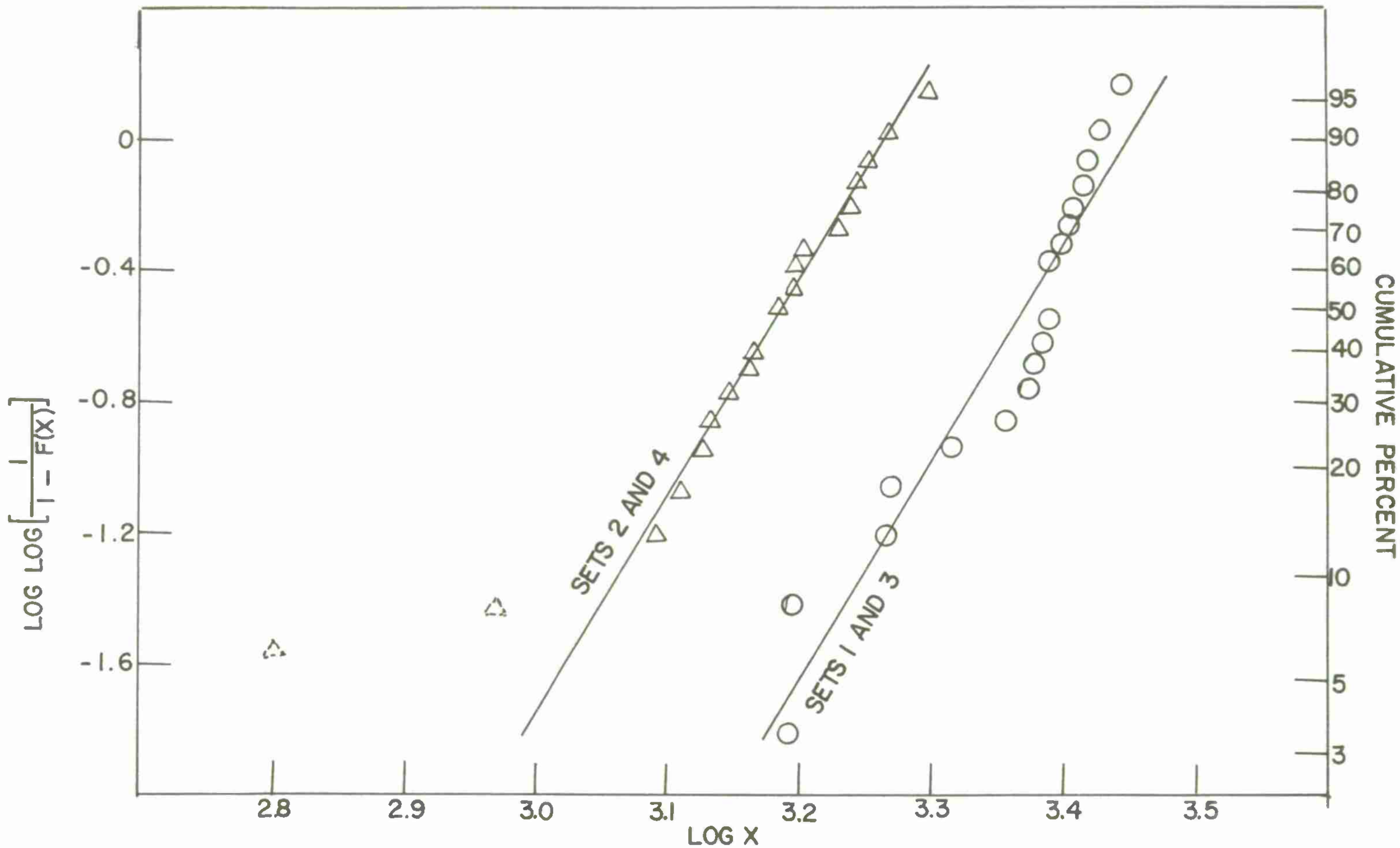

Figure 2. Linear Weibull distribution plots for Versilok 204

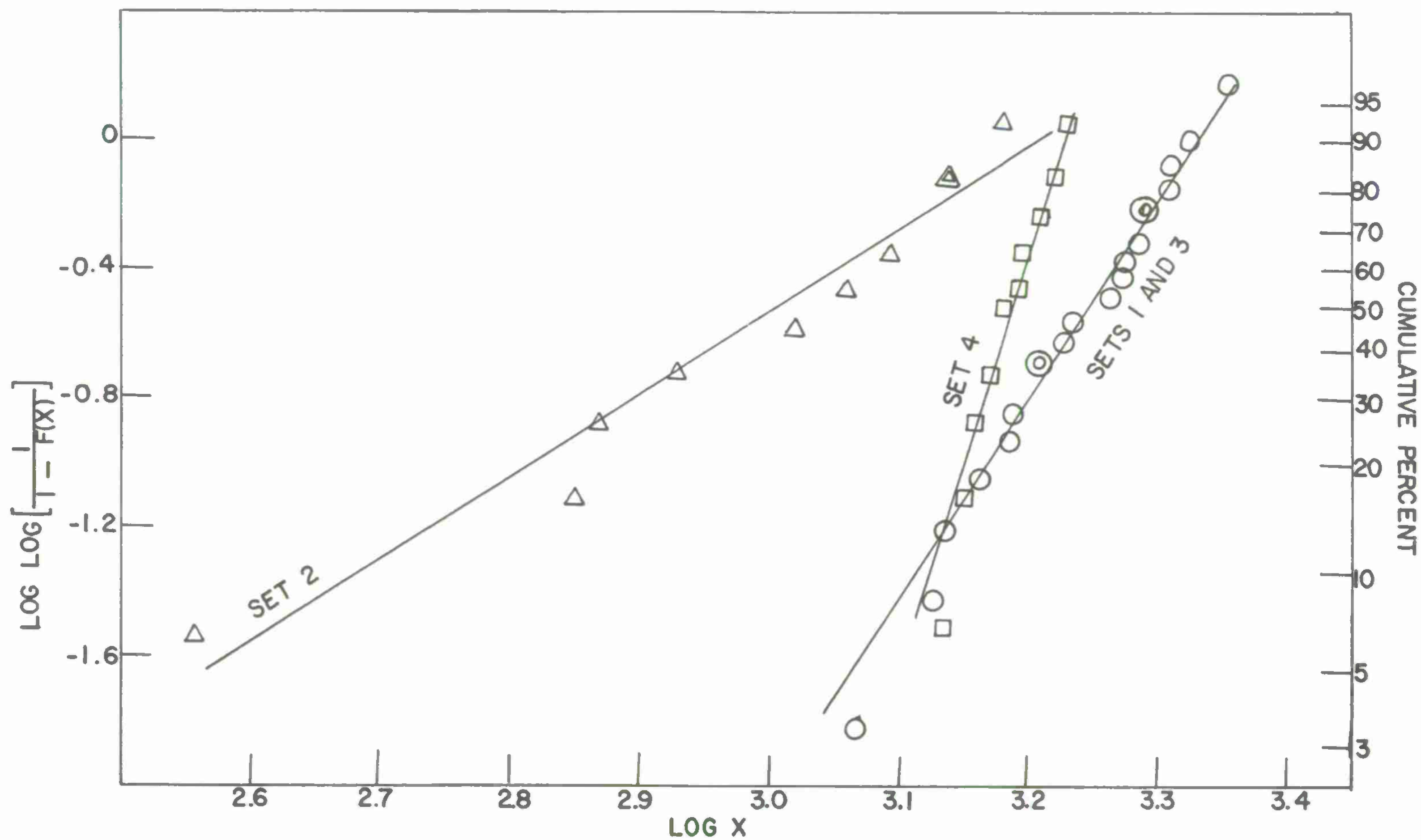

Figure 3. Linear Weibull distribution plots for Loctite Depend

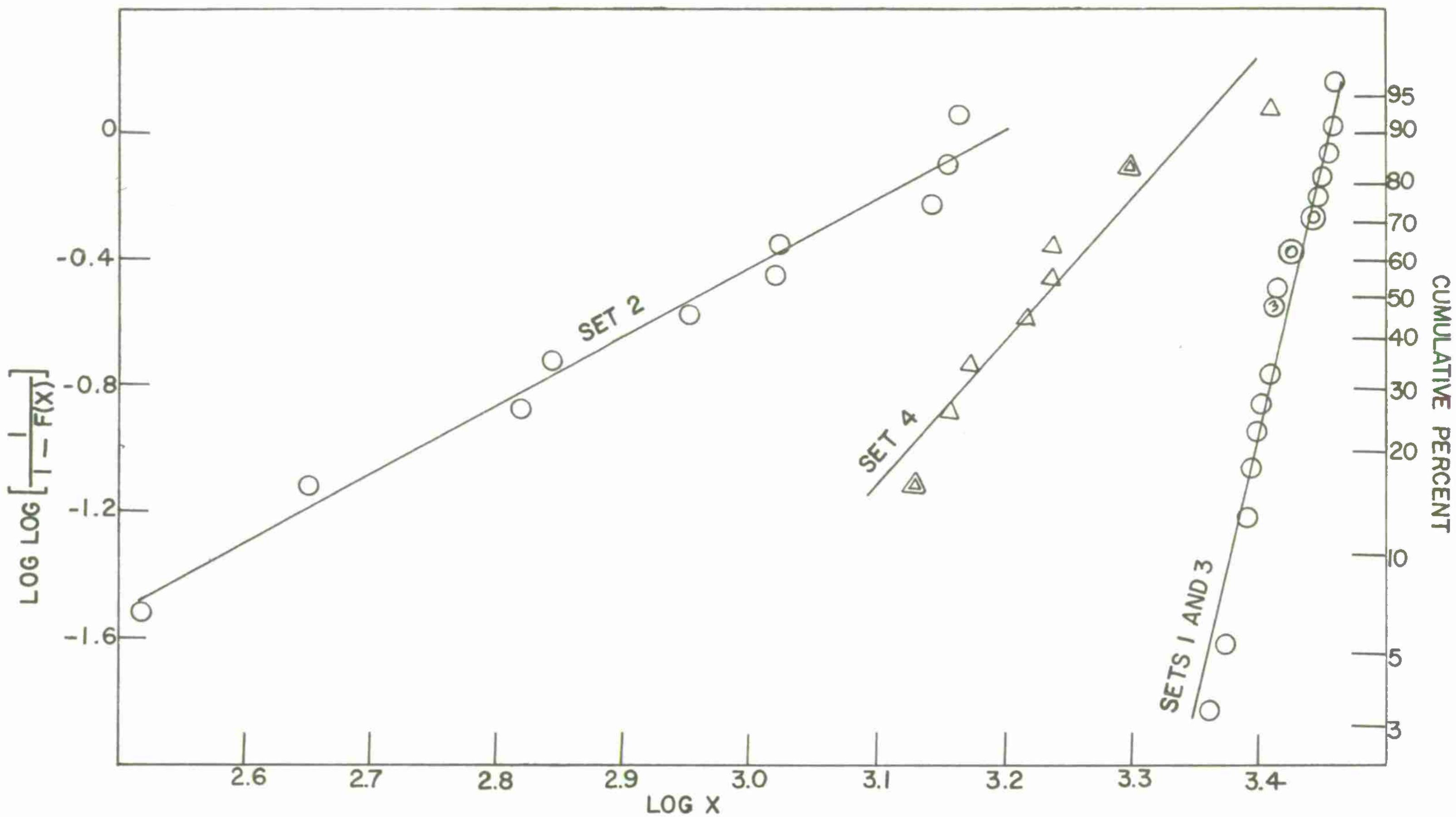

Figure 4. Linear Weibull distribution plots for Dymax 845

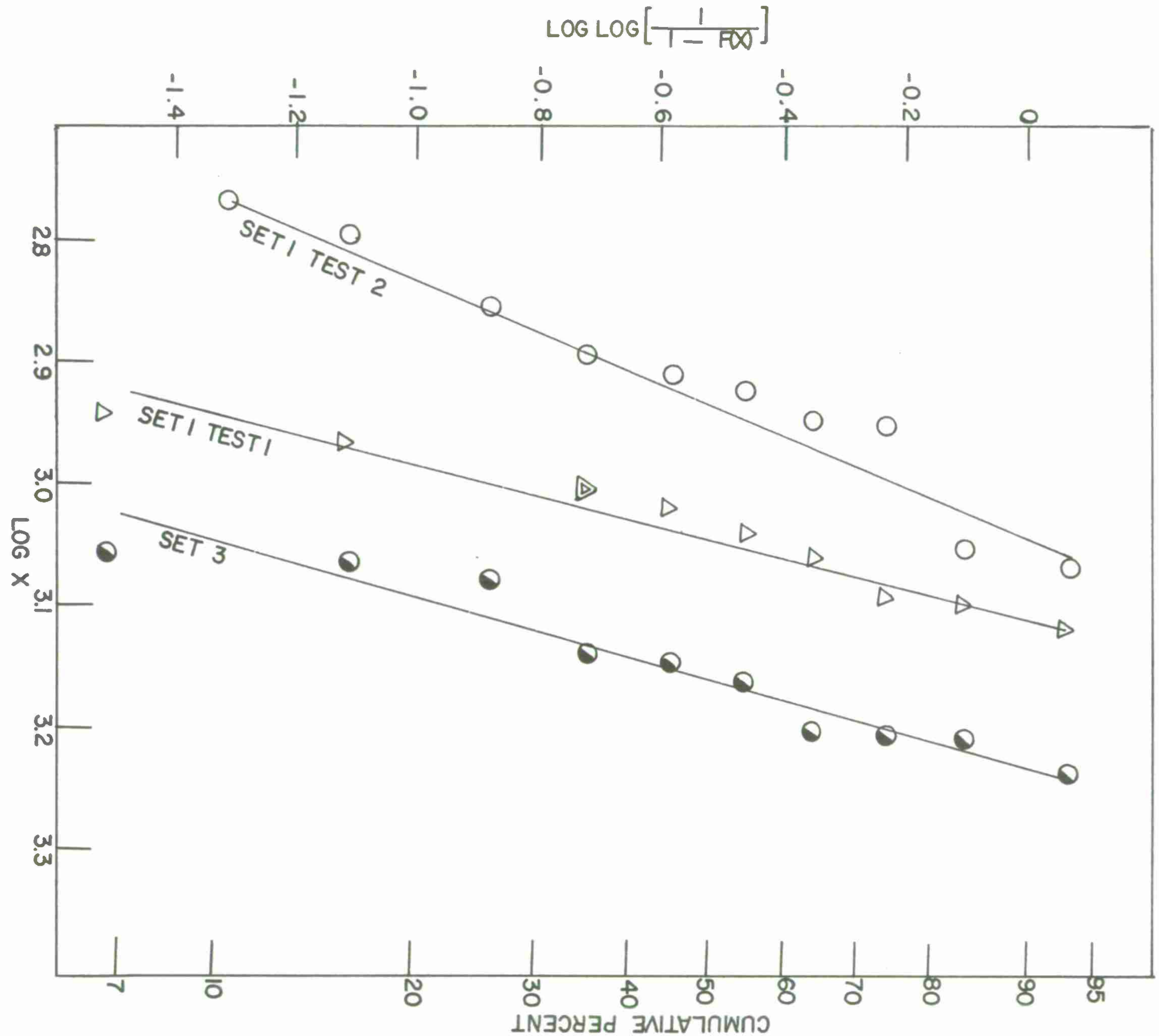

Figure 5. Linear Weibull distribution plots for Versilok 200

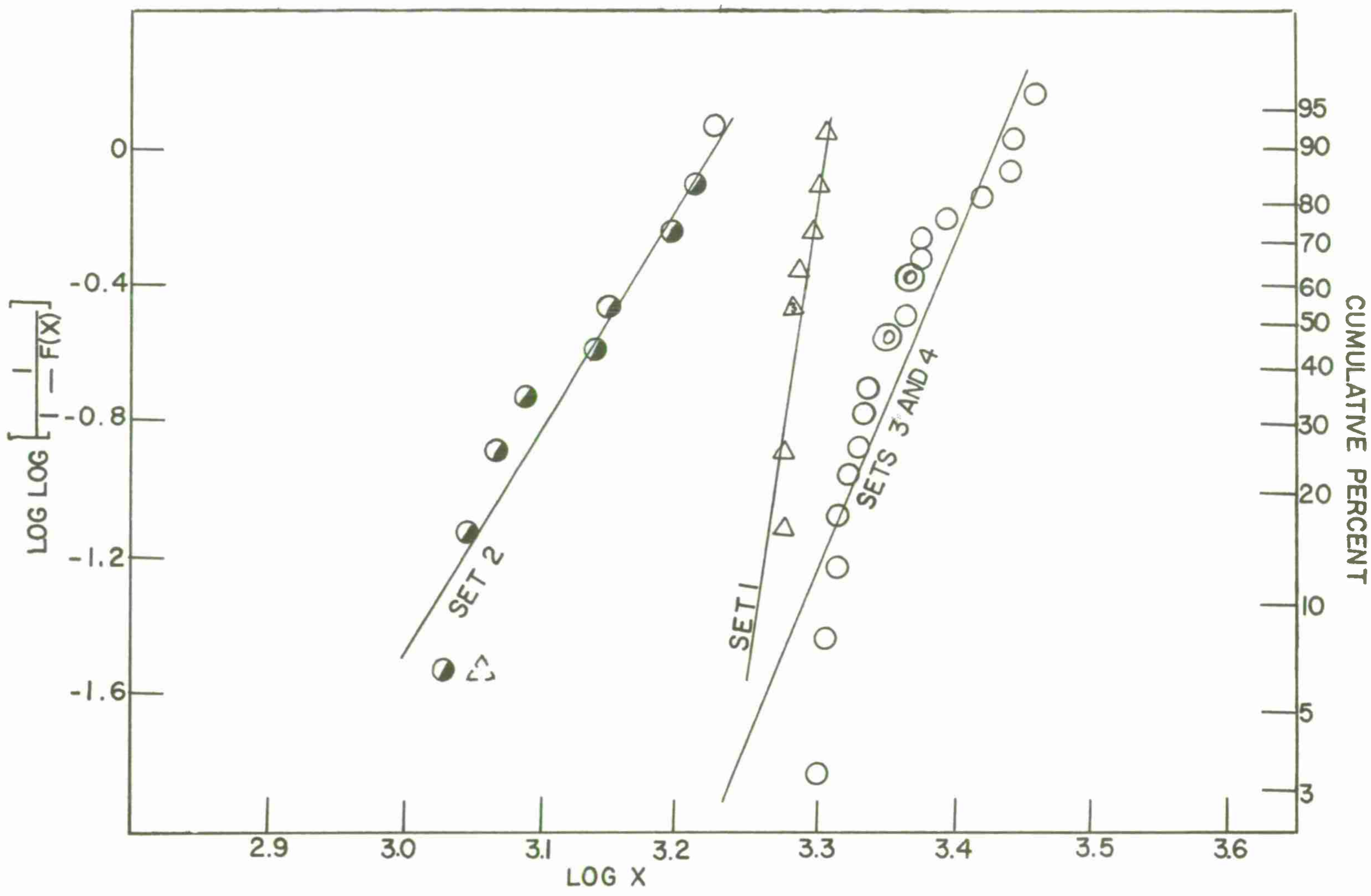

Figure 6. Linear Weibull distribution plots for Plastilock A-1

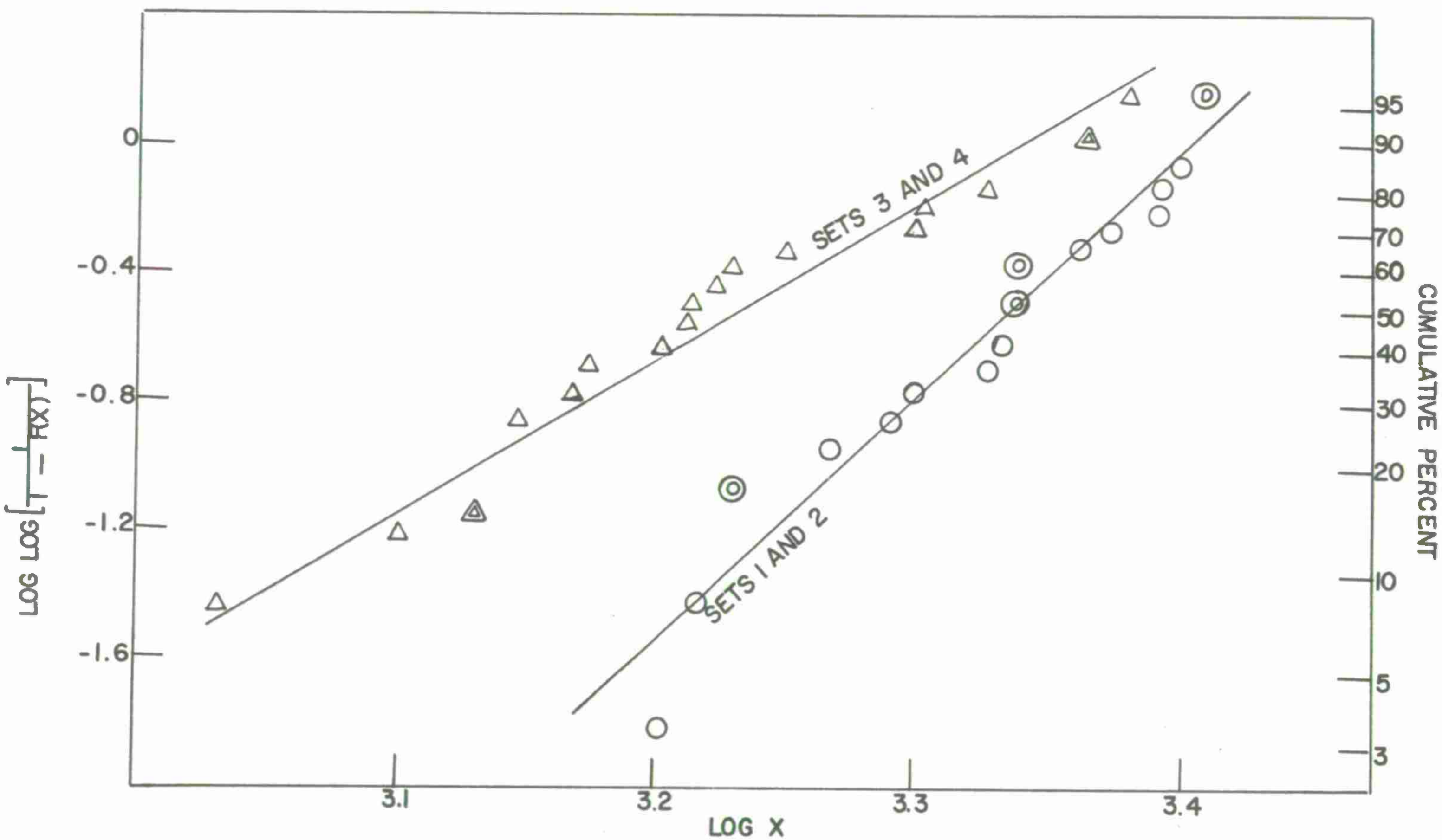

Figure 7. Linear Weibull distribution plots for Epibond 1210

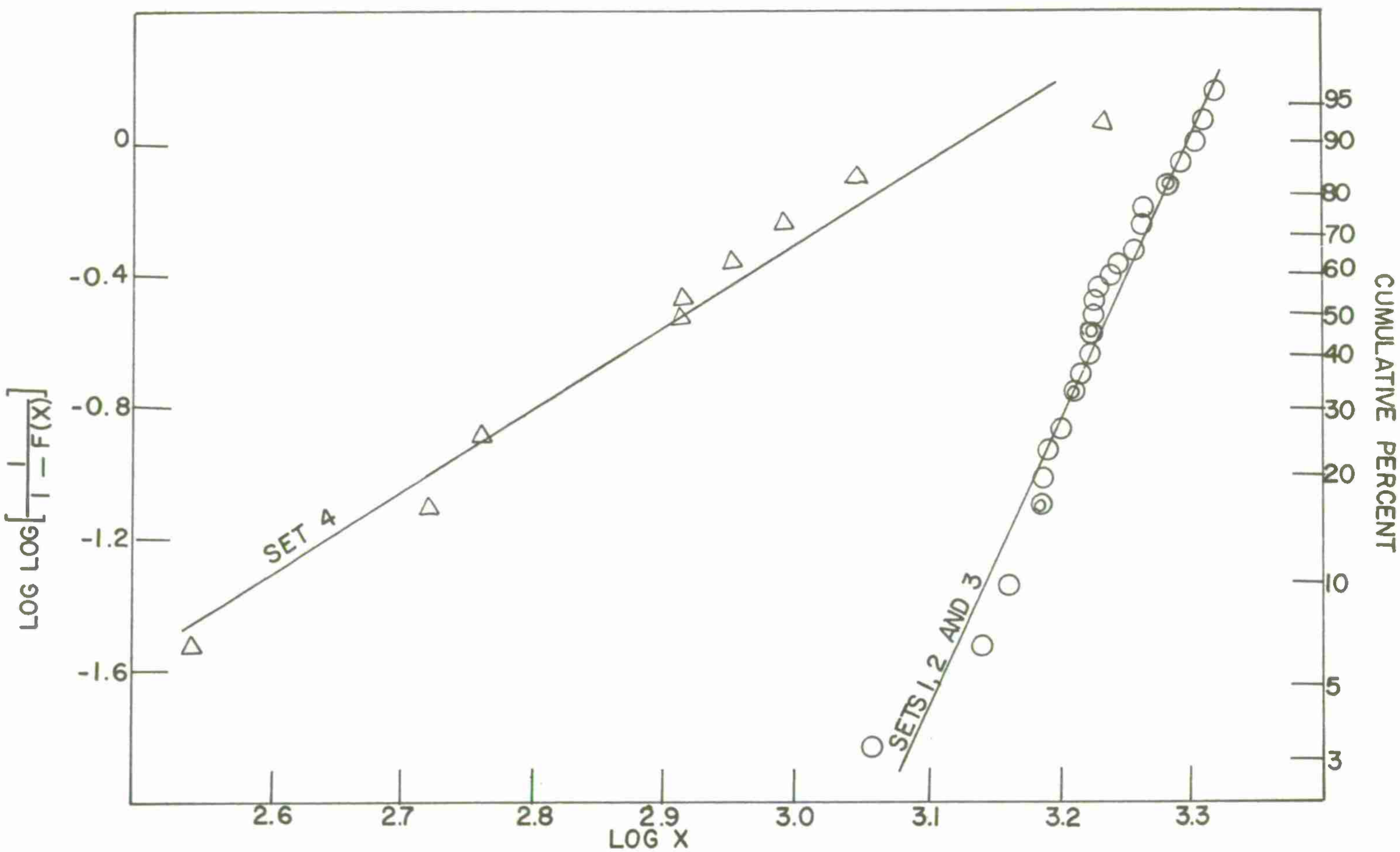

Figure 8. Linear Weibull distribution plots for Epon 828/Epon curing agent V-40

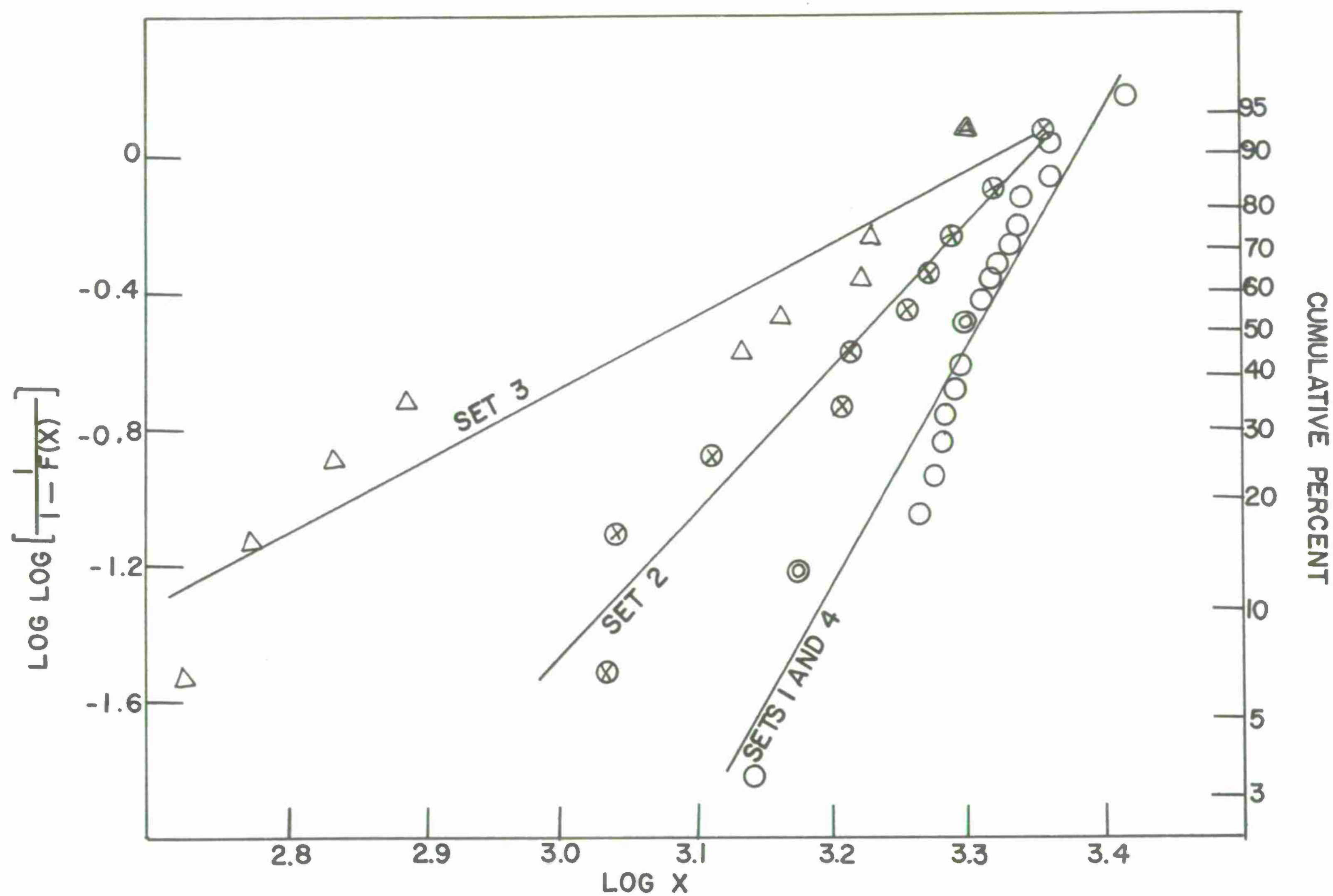

Figure 9. Linear Weibull distribution plots for Cybond 4533

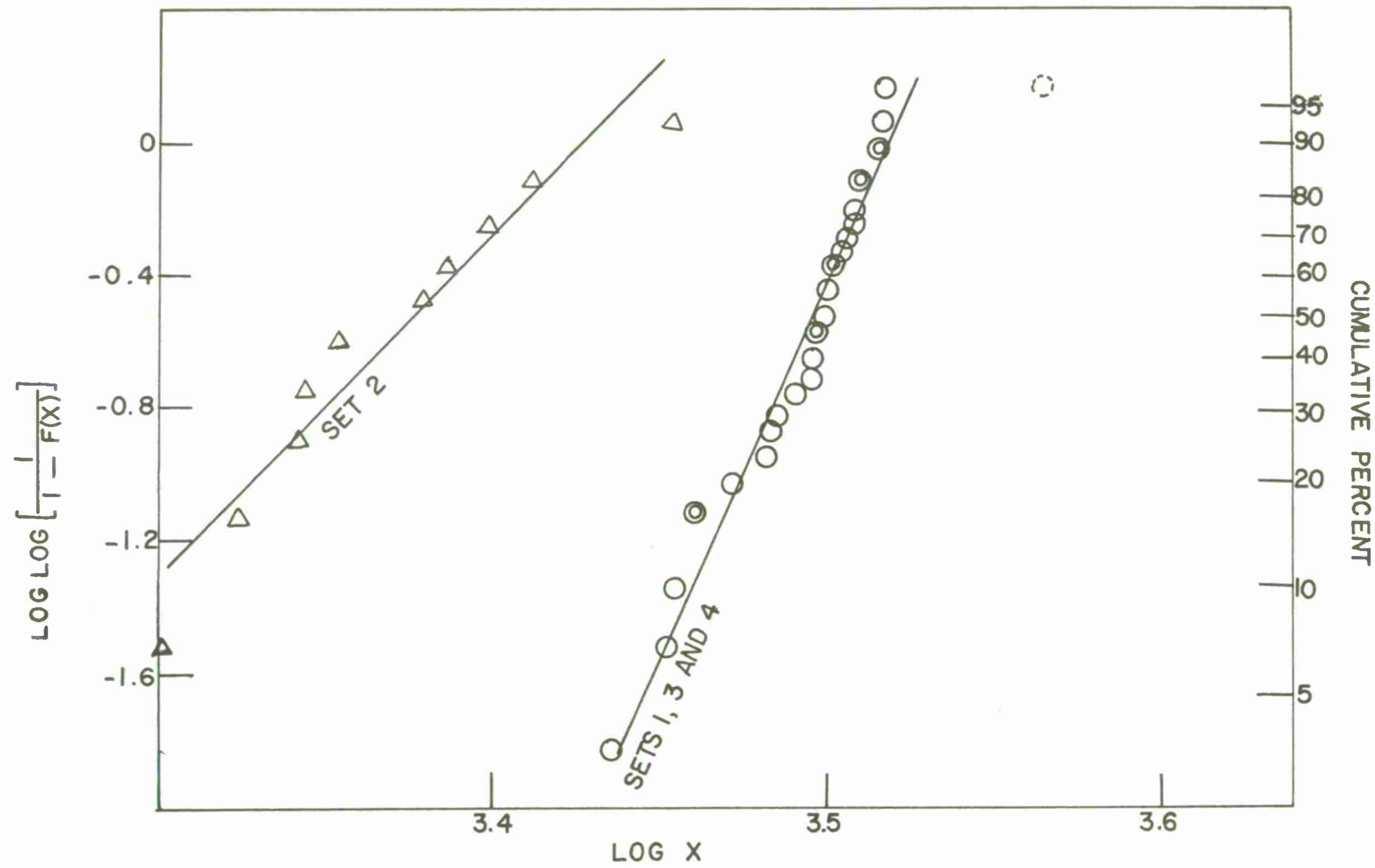

Figure 10. Linear Weibull distribution plots for Conastic 830

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